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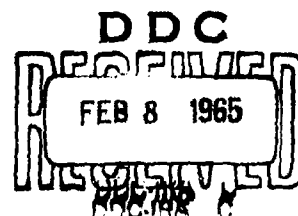
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HOWARD UNIVERSITY

DEPARTMENT OF CHEMISTRY

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THIRD QUARTERLY REPORT
1 October 1963 to 1 January 1964

MICROWAVE SPECTRA AND DIELECTRIC
PROPERTIES OF VARIOUS AZIDES

Contract DA-44-009-AMC-217T

U. S. Army Engineer Research and Development Laboratories
Fort Belvoir, Virginia

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| <p>AD Accession No.</p> <p>Howard University, Washington, D. C.</p> <p>Microwave Spectra and Dielectric Properties of Various Azides. Project No. 8X99-25-001-41</p> <p>C. P. Carter and G. C. Turrell</p> <p>Contract DA-44-009-AMC-217(T)</p> <p>Third Quarterly Rept 1 Oct 63 to 1 Jan 64</p> <p>13 pp 3 Tables Unclassified Rept</p> <p>The dielectric constant of lead azide has been measured in the frequency range 39-49 Kmc.</p> <p>For β-lead azide the real part of the dielectric constant tends to increase with increasing frequency, ranging from about 11 at 39 Kmc to 15 at 49 Kmc. The imaginary part of the dielectric constant shows no clear trend in this frequency range, where its average value is approximately 0.33.</p> | <p>Unclassified</p> <p>1. Azides</p> | <p>AD Accession No.</p> <p>Howard University, Washington, D. C.</p> <p>Microwave Spectra and Dielectric Properties of Various Azides. Project No. 8X99-25-001-41</p> <p>C. P. Carter and G. C. Turrell</p> <p>Contract DA-44-009-AMC-217(T)</p> <p>Third Quarterly Rept 1 Oct 63 to 1 Jan 64</p> <p>13 pp 3 Tables Unclassified Rept</p> <p>The dielectric constant of lead azide has been measured in the frequency range 39-49 Kmc.</p> <p>For β-lead azide the real part of the dielectric constant tends to increase with increasing frequency, ranging from about 11 at 39 Kmc to 15 at 49 Kmc. The imaginary part of the dielectric constant shows no clear trend in this frequency range, where its average value is approximately 0.33.</p> | <p>Unclassified</p> <p>1. Azides</p> |
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Dielectric Constants of the Lead Azides

INTRODUCTION

In a previous report details were given of the method used for measuring the shift in the resonance frequency of a cavity due to the introduction of a small rod of a dielectric material. It was shown how this frequency shift is related to the real part of the dielectric constant of the material.

The method of determining the change in the Q's of the cavity and the relationship between these and the imaginary dielectric constant of the material were also quantitatively given. Experimental data were presented on one sample of polystyrene and one sample of lead azide in the frequency range 39-42 Kmc. The precision of the data was discussed in the case in which the same sample is used throughout a series of measurements.

In this report the data have been extended to include several samples of polystyrene and lead azide in the frequency range 39-49 Kmc. These results indicate the effect of sample shape and irregularity on the precision of the results. It is shown that such factors as (1) the irregular shape of the crystals, (2) the fact that the ends of the sample are not exactly at nodes in the field at all frequencies, (3) the very large number of crystal defects present, etc., have effects on the values of the dielectric constant determined by the present technique.

II. EXPERIMENTAL

Measurements were made on five samples of polystyrene in the frequency range 39-42 Kmc and on four samples of polystyrene in the frequency range 45-49 Kmc. One sample of α -lead azide and four samples of β -lead azide were studied in the frequency range 39-49 Kmc. These azide samples were of various sizes. The masses ranged from 0.05 mg to 1.3 mg with corresponding volumes of 1.0×10^{-5} to $2.7 \times 10^{-3} \text{ cm}^3$. The volumes were calculated using the literature density values as determined by x-ray diffraction and the sample weight. This fact is mentioned because the method does not take into account the presence of crystal defects.

All measurements were taken as described in an earlier report. Each entry represents an average of ten measurements. The TM_{012} mode was used throughout and the width of the resonance curve, Δf_1 , was taken at -7.0 db for the Q measurements in all cases.

Instrumental difficulties have plagued this work continuously. First a ripple in the klystron power supply, causing an unsteady r-f output, foreclosed every attempt at attaining meaningful data. This was followed by trouble with the spectrum analyzer used to put marker on the oscilloscope trace of power versus frequency. In each of these cases unsuccessful attempts were made to correct the malfunctions in this laboratory. Assistance was obtained from the Electronics Service Section of ERDL at Fort Belvoir but the malfunctions could not be isolated and corrected. Finally the power source unit and the spectrum analyzer were returned to the manufacturer, Polcrad, Inc., in New York. After complete overhaul, these instruments are now

functioning properly..

Considerable time and effort were expended in attempts to design and build a shutter which would facilitate and accelerate the Q-measurements. A drawing of one design was shown in one of the earlier reports. Design of such a shutter for a single frequency would not present major difficulties; but a shutter to cover a wide range of frequencies presents problems which, as yet, we have not solved. In every attempt which we have made, either the loss at the shutter was too large or data were not reproducible. While it is thought that the design of such a shutter is quite feasible, it was considered inadvisable to expend more time in this direction at this time.

III. TABULATION OF DATA

The observed data are recorded in tables I-V. In table I are recorded all data necessary for calculation of the real dielectric constant of lead azide. The values of the real dielectric constant ϵ' , calculated from this data are recorded in table II. The corresponding data for polystyrene are recorded in tables III and V.

TABLE I
Summary of Observed Data for Calculation of $\frac{f_o}{f_s}$ of Lead Azide

| Sample # | Type | Weight of Sample (mg) | Volume of Sample (cm ³) | Frequency (Mc) $\rightarrow f_o$ | | | | | | $\frac{f_o}{f_s}$ | | | | | |
|----------|------|-------------------------------|-------------------------------------|----------------------------------|-----------------------|-----------------------|-----------------------|-----------------------|-----------------------|-----------------------|-----------------------|-------------------------|--|--|--|
| | | | | 39.46 | 40.30 | 41.40 | 42.20 | 44.90 | 45.90 | 46.90 | 47.90 | 48.5 | | | |
| | | | | $\frac{f_o}{f_s}$ (mc) | | | | | | | | | | | |
| 1 | CL | 1.273 | 2.674 x10 ⁻³ | 117 | 127 | 125 | 114 | | | | | | | | |
| 2 | CL | 0.1728 | 3.520 x10 ⁻⁵ | 22.4 | 22.8 | 23.7 | 19.1 | 25.4 | 26.0 | 26.2 | 26.7 | 27.5 | | | |
| 3 | CL | 0.0468 | 9.53 x10 ⁻⁶ | 7.9 | 8.8 | 7.5 | 8.9 | 9.1 | 9.6 | 9.9 | 10.6 | 11.5 | | | |
| 4 | CL | 0.0580 | 1.181 x10 ⁻⁵ | 10.0 | 10.4 | 10.9 | 12.5 | | 13.8 | 15.8 | | | | | |
| 5 | CL | 0.0828 | 3.808 x10 ⁻⁵ | 30.6 | 31.0 | 31.5 | 32.9 | 35.4 | 37.7 | 43.6 | 45.5 | 47.1 | | | |
| | | Cavity Volume cm ³ | | 0.4697 | 0.4531 | 0.4344 | 0.4128 | 0.3862 | 0.3794 | 0.3639 | 0.3588 | 0.3516 | | | |
| | | G \rightarrow | | 0.950 | 0.911 | 0.840 | 0.831 | 0.734 | 0.702 | 0.693 | 0.645 | 0.627 | | | |
| 1 | | | | 1.796x10 ³ | 1.694x10 ³ | 1.624x10 ³ | 1.544x10 ³ | | | | | | | | |
| 2 | | | | 1.334x10 ⁴ | 1.287x10 ⁴ | 1.234x10 ⁴ | 1.173x10 ⁴ | 1.097x10 ⁴ | 1.079x10 ⁴ | 1.034x10 ⁴ | 1.019x10 ⁴ | 1.0099 x10 ⁴ | | | |
| 3 | | | | 4.930x10 ⁴ | 4.750x10 ⁴ | 4.560x10 ⁴ | 4.330x10 ⁴ | 4.052x10 ⁴ | 3.981x10 ⁴ | 3.818x10 ⁴ | 3.765x10 ⁴ | 3.689 x10 ⁴ | | | |
| 4 | | | | 4.061x10 ⁴ | 3.850x10 ⁴ | 3.678x10 ⁴ | 3.495x10 ⁴ | | 3.210x10 ⁴ | 3.080x10 ⁴ | | | | | |
| 5 | | | | 1.234x10 ⁴ | 1.190x10 ⁴ | 1.140x10 ⁴ | 1.084x10 ⁴ | 1.014x10 ⁴ | 0.996x10 ⁴ | 0.956x10 ⁴ | 0.942x10 ⁴ | 0.920x10 ⁴ | | | |

f_o = resonance frequency of cavity
 Δf_o = shift in resonance in frequency on introduction of the sample
 V_c = volume of cavity
 V_s = volume of sample
 G = $\frac{f_o^2 d^2}{1.088c^2}$, c = velocity of light, d = diameter of cavity

TABLE II
Summary of ϵ'/ϵ_0 for Lead Azide

| Sample Number | Type | Frequency (Kmc) - f_0 | | | | | | | | | |
|---------------|----------|-------------------------|-------|-------|-------|-------|-------|-------|-------|-------|--|
| | | 39.46 | 40.30 | 41.40 | 42.20 | 44.90 | 45.90 | 46.90 | 47.90 | 48.60 | |
| | | ϵ'/ϵ_0 | | | | | | | | | |
| 1 | α | 6.60 | 6.85 | 7.07 | 6.28 | | | | | | |
| 2 | β | 8.97 | 8.99 | 9.40 | 7.59 | 9.45 | 9.71 | 9.58 | 9.80 | 9.94 | |
| 3 | β | 11.39 | 12.86 | 10.83 | 11.99 | 12.19 | 12.86 | 12.98 | 13.91 | 14.91 | |
| 4 | β | 11.83 | 11.90 | 12.52 | 13.46 | | 15.00 | 16.42 | | | |
| 5 | β | 11.07 | 11.05 | 11.33 | 11.17 | 11.91 | 12.66 | 14.25 | 14.88 | 15.24 | |

TABLE III
Summary of Observed Data for Calculation of ϵ_c of Polystyrene

| Sample Number | Weight of Sample (mg) | Volume of Sample (cm ³) | Frequency (Kc) | | | | | | | | | |
|---------------------------------|-----------------------|-------------------------------------|----------------|--------|--------|-----------------|--------|--------|--------|--------|--------|--|
| | | | 39.46 | 40.30 | 41.40 | 42.20 | 44.90 | 45.90 | 46.90 | 47.90 | 48.6 | |
| | | | | | | Δf (mc) | | | | | | |
| 1 | 0.3330 | 3.250×10^{-4} | 27.7 | 29.5 | 31.4 | 33.0 | | | | | | |
| 2 | 0.6318 | 6.163×10^{-4} | 45.2 | 48.3 | 48.4 | | | | | | | |
| 3 | 0.4398 | 4.219×10^{-4} | 31.0 | 31.6 | 31.6 | 36.4 | | | | | | |
| 4 | 0.3858 | 3.764×10^{-4} | 31.2 | 31.0 | 32.0 | 35.1 | | | | | | |
| 5 | 0.2168 | 2.115×10^{-4} | 18.5 | 17.5 | 18.3 | | | | | | | |
| 6 | 0.1090 | 1.06×10^{-4} | | | | | 24.9 | 24.2 | 24.6 | 27.0 | 24.7 | |
| 7 | 0.0946 | 0.922×10^{-4} | | | | | 24.0 | 24.0 | 27.8 | 28.0 | 28.2 | |
| 8 | 0.1112 | 1.084×10^{-4} | | | | | 17.8 | 17.6 | 20.5 | | 18.6 | |
| 9 | 0.0714 | 0.696×10^{-4} | | | | | 13.0 | 13.7 | 14.0 | 12.7 | | |
| G \longrightarrow | | | 0.950 | 0.911 | 0.840 | 0.831 | 0.734 | 0.702 | 0.693 | 0.645 | 0.627 | |
| Cavity Volume \longrightarrow | | | 0.4697 | 0.4531 | 0.4344 | 0.4128 | 0.3862 | 0.3794 | 0.3639 | 0.3588 | 0.3516 | |

TABLE IV

[illegible]

TABLE V_e''/ϵ_0 of Lead Azide
Summary of Data on

| Sample Number | Type Crystal | Frequency (Kmc) $\sim f_0$ | | | | | | | | | |
|---------------|--------------|----------------------------|-------------------------|-------------------------|-------------------------|------------------------|------------------------|------------------------|------------------------|--|--|
| | | 40.30 | 41.40 | 42.30 | 44.90 | 45.90 | 46.90 | 47.90 | 48.60 | | |
| 1 | α | 4.6 | 4.0 | 5.8 | | | | | | | |
| 2 | β | 1.0 | 1.2 | 0.8 | 1.1 | 1.0 | 1.0 | 1.1 | 1.1 | | |
| 3 | β | 0.3 | 0.3 | 0.2 | 0.3 | 0.2 | 0.3 | 0.2 | 0.3 | | |
| 4 | β | 0.4 | 0.3 | 0.5 | | 0.3 | | | | | |
| 5 | β | 1.3 | 1.3 | 1.2 | 1.0 | 1.3 | 1.2 | 1.2 | 1.0 | | |
| G | | 0.950 | 0.911 | 0.840 | 0.831 | 0.702 | 0.673 | 0.645 | 0.627 | | |
| G f_0 | | 37.49x10 ⁹ | 36.61x10 ⁹ | 34.78x10 ⁹ | 35.07x10 ⁹ | 33.90x10 ⁹ | 32.55x10 ⁹ | 30.99x10 ⁹ | 30.47x10 ⁹ | | |
| v/v_B | α | 0.4790x10 ⁻⁷ | 0.4627x10 ⁻⁷ | 0.4669x10 ⁻⁷ | 0.4403x10 ⁻⁷ | 0.312x10 ⁻⁷ | 3.315x10 ⁻⁷ | 3.276x10 ⁻⁷ | 3.288x10 ⁻⁷ | | |
| 1 | β | 3.558x10 ⁻⁷ | 3.515x10 ⁻⁷ | 3.630x10 ⁻⁷ | 3.445x10 ⁻⁷ | 12.93x10 ⁻⁷ | 12.23x10 ⁻⁷ | 12.10x10 ⁻⁷ | 12.15x10 ⁻⁷ | | |
| 2 | β | 13.15x10 ⁻⁷ | 12.97x10 ⁻⁷ | 13.11x10 ⁻⁷ | 12.93x10 ⁻⁷ | 12.23x10 ⁻⁷ | 12.23x10 ⁻⁷ | 12.15x10 ⁻⁷ | 12.11x10 ⁻⁷ | | |
| 3 | β | 10.83x10 ⁻⁷ | 10.51x10 ⁻⁷ | 10.57x10 ⁻⁷ | 9.965x10 ⁻⁷ | 9.86x10 ⁻⁷ | 9.759x10 ⁻⁷ | | | | |
| 4 | β | 3.294x10 ⁻⁷ | 3.251x10 ⁻⁷ | 3.277x10 ⁻⁷ | 3.094x10 ⁻⁷ | 3.061x10 ⁻⁷ | 3.029x10 ⁻⁷ | 3.040x10 ⁻⁷ | 3.029x10 ⁻⁷ | | |
| 5 | β | | | | | | | | | | |
| e'/e_0 | | | | | | | | | | | |
| 1 | α | 0.220 | 0.185 | 0.249 | 0.255 | | | | | | |
| 2 | β | 0.356 | 0.422 | 0.367 | 0.276 | 0.364 | 0.328 | 0.362 | 0.361 | | |
| 3 | β | 0.395 | 0.380 | 0.262 | 0.258 | 0.367 | 0.245 | 0.263 | 0.243 | | |
| 4 | β | 0.433 | 0.305 | 0.528 | 0.498 | | 0.296 | 0.283 | | | |
| 5 | β | 0.427 | 0.422 | 0.328 | 0.371 | 0.306 | 0.398 | 0.363 | | | |

* $\Delta(\Delta f_0) = \Delta f - \Delta f^0$
 Δf = width of resonance curve at 1/5 incident power level with sample in cavity
 Δf^0 = width of resonance curve at 1/5 incident power level without sample in cavity
 ϵ''/ϵ_0 = relative imaginary dielectric constant

IV. ERRORS

The expression for the real dielectric constant, ϵ'/ϵ_0 , is¹

$$\epsilon'/\epsilon_0 = 1 + \frac{\Delta f}{f_0 G}, \quad V_c/V_s \quad (1)$$

where f_0 is the resonance frequency of the cavity, Δf is the shift in resonance frequency of the cavity on introduction of the sample V_c and V_s are the volumes of the cavity and the sample, respectively, and G is given by

$$G = \frac{1.008 \, c^2}{f_0^2 \, d^2} \quad (2)$$

Here c is the velocity of light, d is the diameter of the cavity, and f_0 has been defined above.

The quantity Δf is read directly from a General Radio Oscillator as described in an earlier report. Frequency calibration of this instrument indicates that it is accurate to 1%.² However the blank spaces in the power versus frequency plot on the oscilloscope screen which serve as markers have a length which is dependent on the amplification in the four-channel amplifier. These factors, along with fluctuation of frequency along the frequency-axis of the oscilloscope, increase the error to about 2-2.5%. A typical series of Δf values are those for sample number 2 lead azide. They are 21.5, 22.2, 22.2, 23.0, 23.0, 22.5, 22.7, 22.0, 22.2 and 22.2 mc with an average value of 22.4 mc. This range of ± 0.5 mc. corresponds to ± 0.2 units of ϵ'/ϵ_0 or about 2% assuming all other errors are negligible.

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1. E. F. Labuda and R. C. LeCraw, Rev. Sci. Instruments 32, 391(1961).
 2. Calibration performed by the General Radio Company.

The quantity f_0 is easily read to 0.01 Kmc which gives 1 part in 4000 in the 40 Kmc region and above. But this is a wave meter reading and is dependent on the Q of the wavemeter cavity. This effect reduces the certainty of f_0 to about 1 in 400 based on the span of the frequency-axis of the oscilloscope.

The size of the cavity is sufficiently large to make the error in its volume small also. Since V_0 is given by $\pi r^2 l$, where r is the radius and the length, its error can be calculated exactly.

$$\Delta V_0 = 2\pi r \Delta r + \pi r^2 \Delta l \quad (3)$$

where ΔV_0 , Δr and Δl are the errors in the volume, radius and length of the cavity, respectively. The cavity was machined such that the error in r and l is about 10^{-3} cm for each. With order of magnitudes of r and l of 0.5 cm and 1.0 cm, respectively, $\Delta V_0 = 4 \times 10^{-3} \text{ cm}^3$. Since V_0 is about 0.5 cm^3 , the error in V_0 is about 0.8%.

The error in V_s , the volume of the sample is not easily estimated. In the milligram range the weight of the samples can be determined to a microgram. This would easily yield about 1 part in 1000 but the density is not known as well. More will be said about this problem below. For the present the error in V_s will be assumed to be of the same order as that in V_0 . Thus it appears that the error in Δf at 2.5% is the largest single error factor and we expect on this basis that the error in $\frac{f}{f_0}$ to be approximately $\pm 3\%$.

V. DISCUSSION

The data on α -lead azide, while limited, tend to confirm our conclusion of the error in this method. This is also true when measurements on the same sample of β -lead azide are considered.

However, when different samples of β -lead azide are compared at the same frequency, the agreement is poor, ranging from about 9-12 at 39 Kmc and about 10-15 at 49 Kmc. Such fluctuations in ϵ'/ϵ_0 are clearly outside the range of experimental error.

As stated earlier a study of several samples at the same frequency affords an opportunity to evaluate the effect of irregularities in the shape of the sample. While the crystals studied appear to be very small parallelepipeds when examined with the naked eye, with the aid of a microscope the faces are observed to be somewhat irregular. This means that instead of dealing with a single crystal, we have a large number of micro-crystals. It may be that the dielectric constant taken along different axes is different, that is to say it may be a tensor instead of a scalar. If this were true, clearly, orientation would greatly affect the values of ϵ'/ϵ_0 observed. Methods to detect this by using a preferred orientation have not been attempted because of the very small size and fragility of these crystals. A tenth of a milligram crystal which is about 4 mm long is not easily oriented, although methods could be devised.

When viewed under a microscope, it is evident that these crystals have many faults and defects. These make one suspicious of the use of the density as determined by x-ray diffraction. It is realized that the use of sample volumes calculated in this manner may

produce fluctuation in ϵ'/ϵ_0 but no other method for determination of densities of these small crystals has been found in the literature or devised by us.

Another factor which should be considered is the fact that the sample increasingly reaches beyond the nodes in the field as the frequency is increased. Although the ends of the sample were positioned at nodes for the lowest frequency, this is true only at that frequency. This effect may be reflected in the increase in ϵ'/ϵ_0 with increasing frequency, although it is more likely to have the opposite effect.

It has been observed that these small crystals will pick up (what has been interpreted as) free surface charges. The effect of this phenomenon, if any, on the experimental results is not known.

Finally, we point out that the rise in ϵ'/ϵ_0 with frequency may be a response similar to that predicted by the results of classical electromagnetic theory.³ On the other hand it could preface the resonance one would expect if the azide ion has a $^3\Sigma$ ground state.⁴

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3. See for example: "Dielectric Materials and Applications", John Wiley and Sons, Inc.
 4. M. Mizushima, Second Quarterly Report, October, 1958-January, 1959, Project Morty, ERDL, Fort Belvoir, Virginia.

VI. PRESENT STATUS

At the present time we are determining ϵ'/ϵ_0 for lead azide in the 50-60 Kmc range. Since the Polarad EHF power source does not come equipped with "plug-in" units above 50 Kmc, it was necessary to design a "dummy" panel and alter the circuit such that beam voltage, reflector voltage, grid voltage, modulation, etc., could be controlled from this panel. In this way we are able to operate a klystron of any frequency range with this supply unit. This is not the most efficient unit however. Frequent interruptions are caused by malfunctioning. The problem has recently been solved by the purchase of an FXR Universal klystron supply. This was thought advisable since the klystron power supply presently being used in the microwave spectrograph does not belong to this project.

Recently, the two matched klystrons (QK-295), which together cover the 50-60 Kmc range, have been giving trouble. They have been returned to the Raytheon Company and as yet no word has been received concerning them. This has forced us to attempt to generate power in this range by using a lower-range klystron coupled to a harmonic multiplier. The frequency of this klystron is such that the second harmonic would cover a large portion of the 50-60 Kmc range. So far we have not been able to detect the second harmonic.

As soon as microwave power is available, the 50-60 Kmc range can be rapidly studied, barring any other serious malfunctioning. Indeed the waveguide circuit is complete up through the 75 Kmc point except for a cavity and the necessary klystrons.